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ISOTOPIC CONSTITUTION OF NICKEL

A DISSERTATION SUBMITTED TO THE FACULTY
OF THE DIVISION OF THE PHYSICAL SCIENCES
IN CANDIDACY FOR THE DEGREE OF DOCTOR OF
PHILOSOPHY

DEPARTMENT OF PHYSICS

1941

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HENRY A. STRAUS

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A New Mass Spectrograph and the Isotopic Constitution of Nickel

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A new double focusing mass spectrograph which has a 90° electric field and a 60° magnetic field has been constructed and used to determine the isotopic constitution of nickel. The instrument and the null method of measurement are described. The isotopic constitution of nickel is found to be Ni^{58} —62.8 percent Ni^{60} —29.5 percent, Ni^{61} —1.7 percent, Ni^{62} —4.7 percent, Ni^{64} —1.3 percent.

INTRODUCTION

THE published values of the abundance ratios among the isotopes of nickel have for some time shown considerable discrepancies. Aston¹ found Ni^{61} to be 1.7 percent of the total and failed to find Ni^{64} . De Gier and Zeeman,² who used the parabola method of positive ion analysis, failed to find Ni^{61} , probably because of the low resolving power of their instrument, but found Ni^{64} to constitute 0.9 percent of the total. Lub,³ using a similar apparatus, also found Ni^{64} to be 0.9 percent of the total, but found Ni^{61} , which he estimated to be 0.1 percent of the total. Dempster⁴ published a photograph of the mass spectrum of nickel in which it appeared that the abundance of Ni^{61} was approximately equal to the abundance of Ni^{64} .

These results were all obtained from photographic observations. Dempster used an oscillating spark between nickel electrodes as an ion source. The others all used a discharge tube containing nickel carbonyl. The organic compounds of the C_5 group, present in the latter case, may lead to erroneous estimates of the relative intensities of the nickel lines by their superposition on the nickel lines.

APPARATUS

A new double focusing mass spectrograph has been constructed. The design of this instrument is based on the theory published by R. Herzog.⁵ A radial electric field is used as an energy analyzer in this instrument. The mean orbit radius in this field is ten cm and the deflection of the beam is 90° . Guard plates, which are applied at the ends of the field to reduce the stray field, are cut so that they do not interfere with the passage of the beam. A magnetic field serves as a momentum analyzer and gives the mass dispersion. The mean radius of the orbits in this field is 16.6 cm and the beam is deflected 60° .

In Herzog's paper the fundamental focusing condition is given by

$$(l' - g')(l'' - g'') = f^2. \quad (1)$$

Here l' is the distance from the source slit to the edge of the field, measured along the orbit, and g'' is the distance beyond the field at which an originally parallel bundle of rays meet; l'' is the distance beyond the field at which the orbits intersect or focus. g' is the distance from the field to the point from which diverge orbits that are parallel after passing through the field. f is the focal length. Continuing in Herzog's notation, we

¹ F. W. Aston, Proc. Roy. Soc. **149**, 396 (1935).

² J. de Gier and P. Zeeman, Proc. K. Akad. Amst. **38.8**, 810 (1935).

³ W. A. Lub, Proc. K. Akad. Amst. **42.3**, 253 (1939).

⁴ A. J. Dempster, Phys. Rev. **50**, 98 (1936).

⁵ R. Herzog, Zeits. f. Physik **89**, 447 (1934).

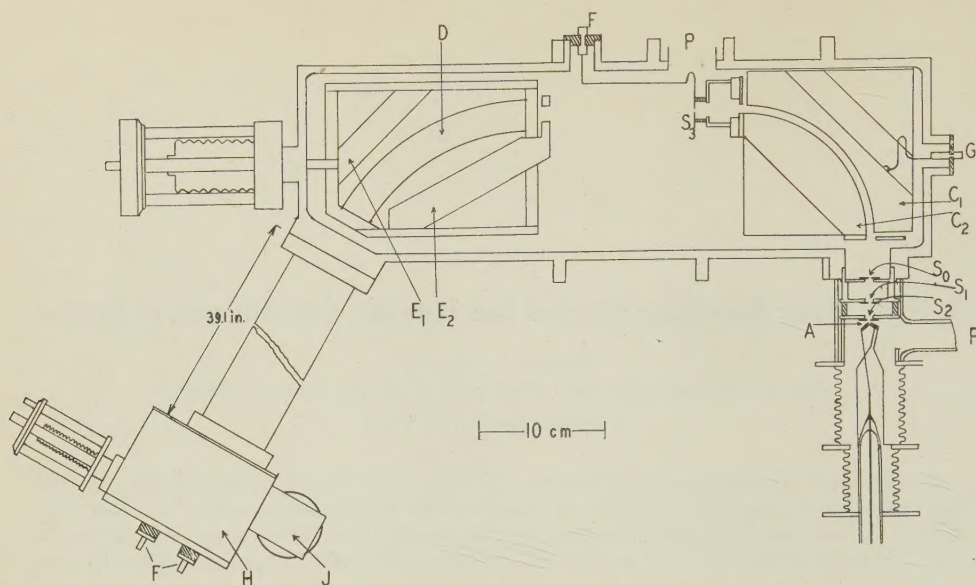


FIG. 1. Plan of mass spectrograph including section through deflection chamber and source. The yoke and core of the magnet are omitted. The parts are labeled as follows: S_0 , S_1 , S_2 , S_3 are slits. P refers to pumping lines. A is the spark and C_1 and C_2 are the electric field plates. G is the lead for the deflecting potential. F are electrometer leads. D is the pole piece and E_1 and E_2 the pole piece spacers. H is the collector chamber and J the secondary electron magnet.

have for the radial electric field,

$$f_e = a_e / \sqrt{2} \sin \sqrt{2} \Phi_e, \quad (2)$$

$$g_e = a_e \cot \sqrt{2} \Phi_e. \quad (3)$$

In these equations a_e is the radius of the orbit in the electric field and Φ_e is the deflection in the field. For the dimensions chosen, f_e is 8.89 cm and g_e is -5.38 cm. The source slit, S_0 in Fig. 1, is at a distance 3.50 cm from the entrance to the electric field. Equation (1) thus shows that the image produced by the electric field is at $l'' = 3.5$ cm. In the magnetic field the corresponding equations are

$$f_m = a_m / \sin \Phi_m, \quad (4)$$

$$g_m = a_m \cot \Phi_m, \quad (5)$$

where a_m is the orbit radius in the magnetic field and Φ_m is the angle of deflection. For the dimensions given, f_m is 19.17 cm and g_m is 9.58 cm.

To obtain velocity focusing, it is necessary that the velocity dispersion in the two fields be equal. For the electric field this quantity is measured by $K_e = a_e [1 + f_e / (l_e' - g_e)] = 20$. Thus $K_m = a_m [1 + f_m / (l_m'' - g_m)] = 20$. This fixes l_m'' , the distance from the magnetic field to the collector, as 103.1 cm. The fundamental equation

for a focus now determines the last dimension of the apparatus.

When g_m , l_m'' and f_m are substituted in $(l_m' - g_m)(l_m'' - g_m) = f_m^2$, l_m' , the distance from S_3 to the magnetic field is 13.5 cm. The dispersion, as calculated from Herzog's theory, is 4.9 mm for 1 percent mass difference, agreeing approximately with 5.05 mm observed.

As mentioned above, the electric field forms an energy spectrum 3.5 cm beyond the field. A two-millimeter slit, S_3 is placed here to select a range of energy from the ion beam to be passed into the magnetic field. At a distance of 13.5 cm beyond this slit the ions effectively enter the magnetic field. The effective magnetic field was used in calculating the orbits. The effective field includes the integrated effect of the stray field which was calculated by the Schwartz transformation. The final image is formed 103 cm from the point at which the ions effectively leave the magnetic field.

In the instrument, as used, the source slit, S_0 , was 0.15 mm wide. Between it and the source were two other slits. S_1 was 2 cm from S_0 and was 2.0 mm wide. It was at ground potential with S_0 . S_2 was just in front of the spark A , and was maintained at a positive potential with

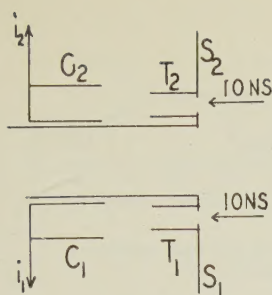


FIG. 2. The collector system in cross section. S_1T_1 and S_2T_2 are the shields and C_1 and C_2 are the collectors. The charges collected are measured by the circuit indicated in Fig. 3.

the source. This slit system greatly impeded the flow of gas from the source into the spectrograph chamber. Pumping the source independently of the chamber thus made it possible to maintain a good vacuum while the source was running.

The ions to be studied were produced in a spark between electrodes of the metal. The spark was maintained at a positive potential with respect to ground by a half-wave rectifier set with condensers whose potential was indicated by a resistance and a millimeter. The spark was energized by a Tesla circuit.

The potential applied to the plates of the electric field was supplied by a high voltage lead battery on trickle charge in some runs and by a stabilized source⁶ of 950 volts for the rest of the observations.

The magnet consisted of a pair of pole pieces separated by 0.157 inch which were mounted as a unit inside the spectrograph chamber and an external double yoke on which were mounted the core pieces. Each core piece was wound with 750 turns of number twelve wire. Between alternate layers of the windings, sheets of copper with exposed fins were interposed to reduce the time required for the magnet to come to thermal equilibrium. The magnet current was observed by means of a potentiometer. The magnet current, which was controlled manually with slide wire rheostats, was supplied by a twelve-volt lead battery.

The chamber which held the field assemblies was a bronze casting which had been dipped in tin to make it vacuum tight. The lid was removable to permit access to the field assemblies. Steel inserts were included in the top and the bottom of the chamber to reduce the power

required to attain the magnetic field intensity required and to reduce the stray field.

The collector system, whose essential features are shown in Fig. 2, consisted of a pair of Faraday chambers, C_1 and C_2 , connected through amber bushings to the electrometers. The shields were equipped with tubes, T_1 and T_2 , back of the openings, which were somewhat wider than the focused beam of an isotope. The whole assembly was placed in a magnetic field so the secondary electrons from the openings would not reach the collectors. One of the collectors was fixed in position. The other could be varied in position, which could be observed, by a micrometer screw acting through a sylphon bellows.

Each collector was connected to an electrometer and to one side of a condenser whose other plate was mounted on split insulation and connected to a source of potential, as in Fig. 3. As the collectors receive charge, the two electrometers are maintained at zero potential by adjusting R_1 , R_2 , and R_3 . R_1 and R_2 are dial boxes and R_3 is a potentiometer. After charge has been collected, the ratio of the charges on the two systems when the electrometers show a null reading can be found from the equation

$$(Q_1/Q_2) = kR_1/(R_1 + R_2). \quad (6)$$

Here $k = C_1/C_2$ and is determined by a capacity bridge measurement. The method is similar to that developed by V. A. Bailey⁷ and Vanderberg.⁸

OBSERVATIONS

The observations on the abundance ratios in nickel were made with the null method described

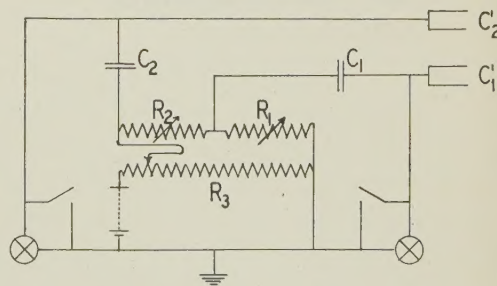


FIG. 3. The circuit used to make the charge comparisons. C_1' and C_2' are the collectors.

⁶ R. D. Evans, Rev. Sci. Inst. 5, 371 (1934).

⁷ V. A. Bailey, Phil. Mag. 50, 381 (1929).

⁸ R. M. Vanderberg, Phys. Rev. 59, 114A (1941).

above. The pressure in the spectrograph, as indicated by an ionization gauge, was less than 5×10^{-6} mm during all the final measurements and was usually about 1 or 2×10^{-6} mm. These pressures were maintained to avoid excessive scattering and loss of charge in the ion beams. The magnet and the voltage stabilizer were allowed to come to equilibrium before any readings were taken.

The collectors were set to a separation corresponding to two mass units to obtain the ratios $\text{Ni}^{58} : \text{Ni}^{60}$, $\text{Ni}^{60} : \text{Ni}^{62}$, $\text{Ni}^{62} : \text{Ni}^{64}$. The background was measured by comparing the background at mass numbers 59 and 63 with the current at Ni^{61} . With the collectors set at a separation corresponding to three mass numbers, the ratio $\text{Ni}^{61} : \text{Ni}^{64}$ was obtained, as well as a comparison of the background at mass numbers 59 and 65 with Ni^{62} .

Readings were taken of the ratio of the charges collected by the two electrometers at each of a series of different positions as the beams were moved simultaneously by small steps across the respective collectors. Figure 4 shows the behavior of the ratio during a set of observations. On the plateau the ratio remained constant within a few percent. The ratio observed was found to be insensitive to variations of the electric deflecting field. This indicates that there was no systematic weighting of the ratios due to a partial separation of the isotopes in the electric field, caused by a stray magnetic field.

The means of the measurement are:

$$\text{Ni}^{60} : \text{Ni}^{58} = 0.470 \pm 0.020;$$

$$\text{Ni}^{62} : \text{Ni}^{60} = 0.158 \pm 0.007;$$

$$\text{Ni}^{64} : \text{Ni}^{62} = 0.272 \pm 0.01;$$

$$\text{Ni}^{64} : \text{Ni}^{61} = 0.662 \pm 0.015.$$

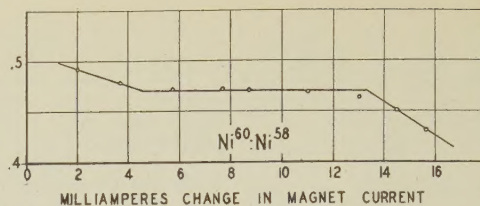


FIG. 4. Results of a set of observations on the ratio $\text{Ni}^{60} : \text{Ni}^{58}$.

Each of these values is the average of approximately 100 electrometer comparisons.

The correction for background is important only for the weak isotope at 61 which is adjacent to the very strong line at 60 and to the strong line at 62. The corrections to 61 were obtained from the measured background at 59 and 63 by assuming that the background at these mass numbers could be ascribed to the neighboring isotopes in the ratio of their abundance. The measured backgrounds were: $59 : \text{Ni}^{61} = 0.20$; $59 : \text{Ni}^{62} = 0.09$; $63 : \text{Ni}^{61} = 0.06$; $65 : \text{Ni}^{62} = 0.007$. With the background correction the ratio $\text{Ni}^{64} : \text{Ni}^{61}$ is 0.745. The isotopic constitution of nickel calculated on this basis is: Ni^{58} —62.8 percent, Ni^{60} —29.5 percent, Ni^{61} —1.7 percent, Ni^{62} —4.7 percent, Ni^{64} —1.3 percent.*

The author wishes to express his appreciation to Professor A. J. Dempster, who suggested this problem, for his advice concerning this investigation.

* Note added in proof.—This isotopic constitution, combined with the isotopic weights published by T. Okuda, K. Ogata, H. Kuroda, S. Shima and S. Shindo, Phys. Rev. 59, 104 (1941), gives 58.84 as the chemical atomic weight of nickel.





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